PROJECT TITLE : ANALYTICAL INVESTIGATIONS

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METHOD DEVELOPMENT

New Extraction Method (YVG)

In order to improve product characterization, a new extraction method was investigated. The method was previously presented to obtain quantitative essential oil extraction of flowers and hops (1).

The method is based on azeotropic steam distillation in conjunction with continuous extraction using a small and constant amount of a water-immiscible solvent (minimum volume of 1.0 ml) possibly containing a suitable internal standard. The new extraction procedure avoids the loss of compounds associated with solvent drying and concentration steps inherent to usual extraction techniques.

Figure 1 shows the micro steam-distillation-extraction apparatus used. The sample (liquid or solid) is placed together with water in flask A, the organic solvent (density > water) in flask B. Dichloromethane was chosen as an organic solvent for our experiments. Both flasks were heated and the water and dichloromethane refluxed. Sample components were steam-distilled from A to C and extracted from the vapour phase and the liquid phase by the organic solvent. Depending on the sample size the extraction time was between 1 and 1.5 hours. The concentrated extract can be used directly for GC or GC/MS analysis.

In the context of trouble-shooting problems, several applications were investigated: profiling of tobacco flavours (Fig. 2), profiling of components in RL samples of different quality (Fig. 3) and characterization of cigarette supplies (inks, varnishes, tipping paper).

A PME Analytical Method is presently being written.

Fatty Acid Analysis

- A GC method for the analysis of acetic, propionic and butyric acid in NOD samples by direct sample injection after dilution of the samples and addition of an internal standard was developed (ETL).

- The recovery of acetic, propionic and butyric acid on steam distillation from liquid samples was checked using (^{14}C) acetic, propionic and butyric acid.

Recoveries were improved to > 90% by addition of inorganic salts (MUM).

Nicotine Analysis

A simple method for the quantitation of nicotine in tobacco samples by GC^2 headspace was worked out (FMO).

SERVICE FOR OTHER GROUPS

- HPLC monitoring of lactic acid in tobacco extracts was re-checked and the method applied to the analysis of 17 RL samples of Project TASK (JJP).
- Lactic and sorbic acid content of another 40 RL samples is presently being determined by HPLC (JJP).
- NO was simultaneously determined in the mainstream and sidestream smoke of 4 experimental cigarettes submitted by Biotechnology. (JJP) (2).
- Headspace analyses were carried out on 9 MFUK and UK2FI flavour samples at the request of QC (FMO) (3) (4).
- 7 cigarette samples (5) and 40 RL samples of Project TASK were analysed by GC^2 for carboxylic acid content (ETL).
- 3 Merit and 2 MLF cigarette samples were analysed by headspace and by GC^2 for fatty acids at the request of Product Development (ETL/FMO) (6) (7).
- 7 Leaf tobacco samples were analysed for minor tobacco alcaloids at the request of SOTA (FMO) (8).
- Several triacetin samples were analysed for QC by GC^2 (YVG).
- Phosphate/sulfate determinations were performed on 14 RL and RCB samples at the request of Product Development (ETL).
- The SH index of 16 TLA cigarette samples was determined (MUM).

REFERENCES

(1) Godefroot-M. et al., J. Chromatography 203 (1981) 325.
(2) Memo from Piadé-J.-J. to Bindler-G., March 18, 1982.
(3) Memo from Bogers-A.-W. to Boder-J.-B., March 17, 1982.
(4) Memo from Fink-W. to Lopes-F., March 17, 1982.
(5) Memo from Murray-M. to Fink-W., March 16, 1982.
(6) Memo from Nagel-P. to Murray-M., March 10, 1982.
(7) Memo from Murray-M. to Nagel-P. and Fink-W., March 16, 1982.
(8) Letter from Corbaz-R. to Moser-F., February 9, 1982.

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FIGURE 1

Micro steam distillation-solvent-extraction apparatus.

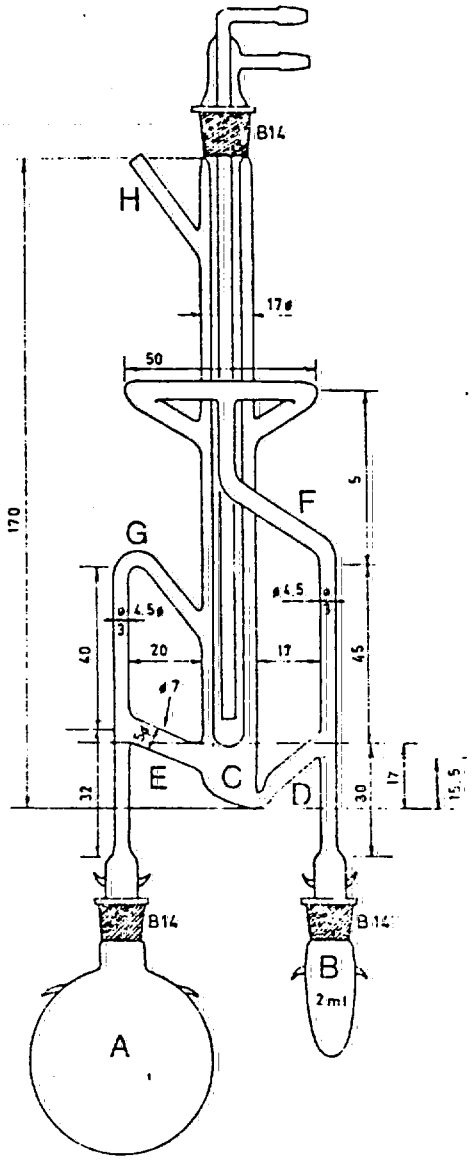

FIGURE 2

Comparison of four types of tobacco by GC^2/micro-steam distillation-extraction.

Amount tobacco extracted : 14g; organic solvent: CH_2Cl_2; extraction time: 1.5hrs.

Chromatographic conditions : Fused silica capillary column 25m x 0.3mm, Superox 0.1; temperature 60° - 200°C.

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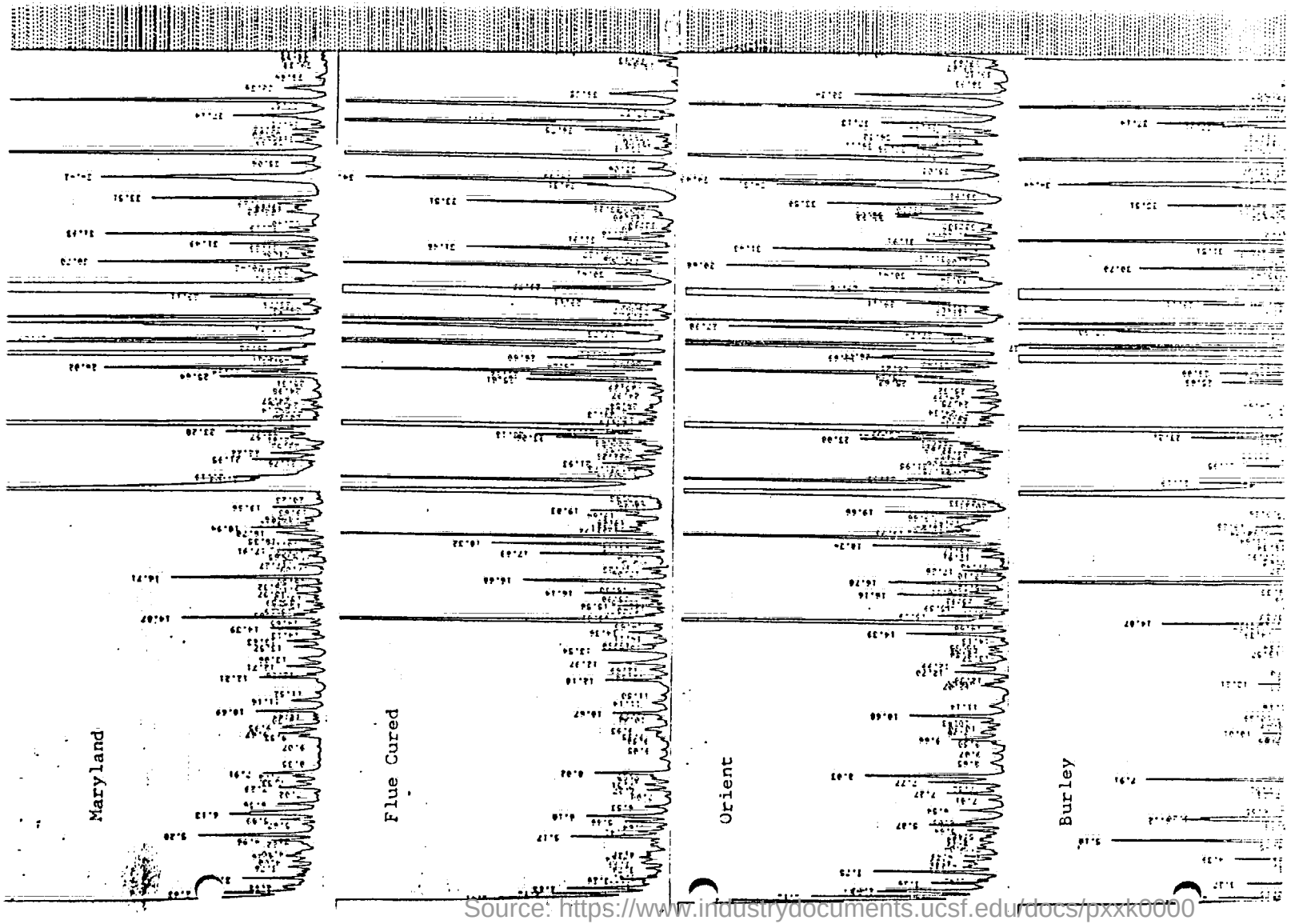

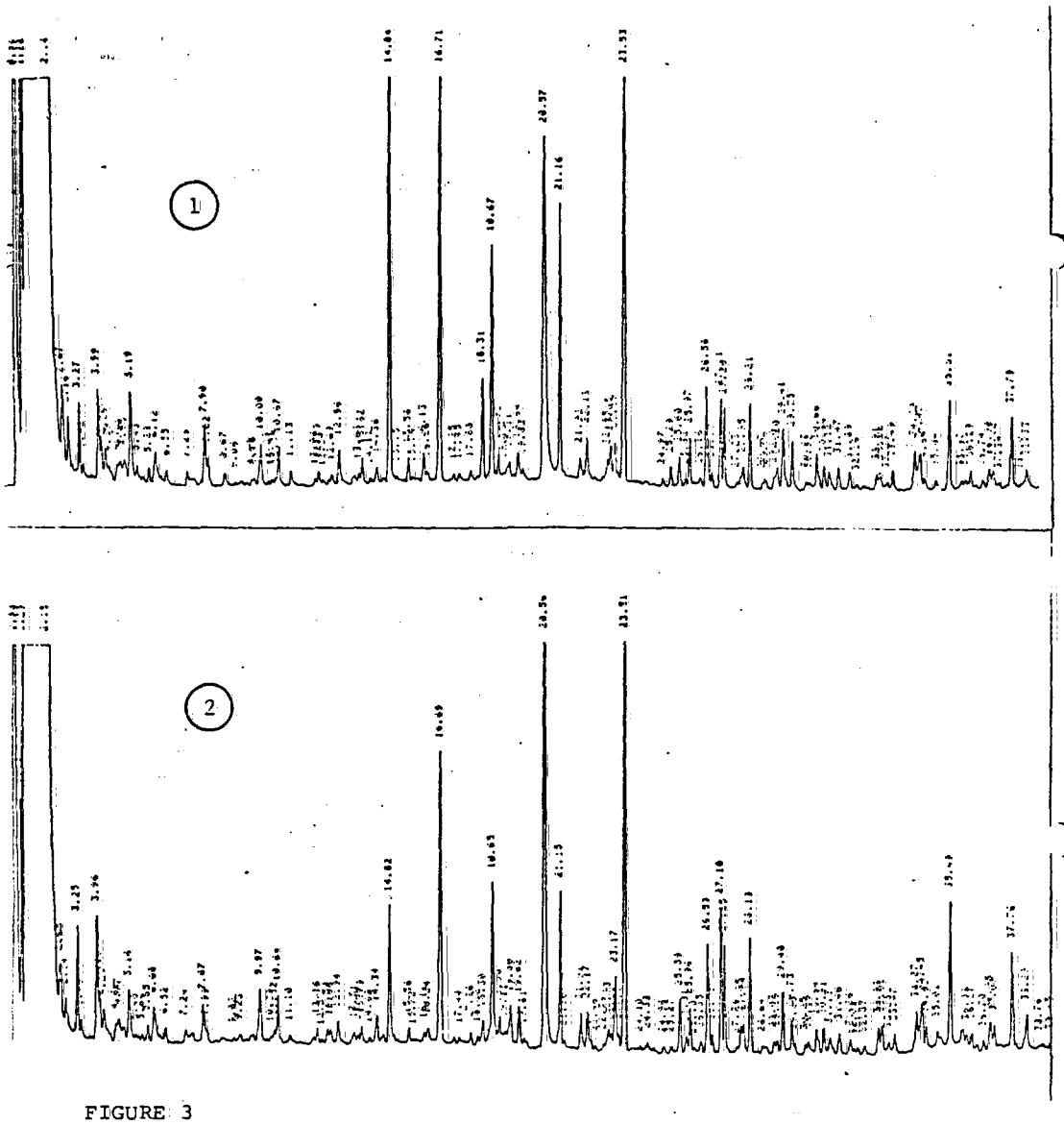

FIGURE 3

Gas chromatogram (GC^2) of extracts obtained by micro steam distillation-solvent-extraction of accepted (1) and rejected (2) RL samples.

Amount RL extracted : 9g; organic solvent : CH_2Cl_2; extraction time; 1.5hr.

Chromatographic conditions as for Figure 2.